PICATINNY ARSENAL
TECHNICAL DIVISION

TECHNICAL REPORT

SUBJECT: A MICROSCOPIC METHOD FOR THE DETERMINATION OF THE PARTICLE (CRYSTAL) SIZE DISTRIBUTION OF "2-MICRON" RDX

PROJECT NO. EPO-AP-13 **REPORT NO.** 1

PREPARED BY: Joseph W. Lavitt **DATE:** 16 February 1953

P. A. SERIAL NO. 1909 **COPY NO.** 29

PRODUCTION ENGINEERING ON STANDARD ARTILLERY AMMUNITION, PROPELLENT POWDER, IGNITION SYSTEMS, TESTING TECHNIQUES

A Microscopic Method for the Determination of the Particle (Crystal) Size Distribution of "2-Micron" RDX

Project No. EPO-AP-13 Report No. 1

Picatinny Arsenal Technical Report No. 1909 16 February 1953

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Agency Performing Work:	Picatinny Arsenal, Dover, New Jersey
Agency Authorizing Work:	ORDIM
Project No.:	EPO-AP-13
DOA Priority Designation:	None
Project Title:	Production Engineering on Standard Artillery Ammunition, Propellent Powder, Ignition Systems, Testing Techniques

OBJECT

To develop a method for the particle (crystal) size distribution of RDX having a nominal diameter of 2 microns.

SUMMARY

A microscopic method for the determination of particle (crystal) size distribution of "2-micron" RDX has been developed. It involves the measurements of 200 crystals of RDX with a microscope equipped with a Filar type micrometer eyepiece. The data is assembled and tabulated in accordance with a simplified statistical technique for group data. The average particle size and standard deviation are then calculated from the group data by means of a few convenient mathematical operations. The average particle size represented by the value $\bar{X}$ and the standard deviation together give a mathematical representation of the particle size distibution. The particle size distribution of the "2-micron" RDX used in this work is represented by the average particle (crystal) size of 2.83 microns with a standard deviation of 1.30 microns. It is estimated that approximately one hour working time is required for one (1) determination.

The applicability of the Andreasen Pipette Method to the determination of the particle (crystal) size determination of "2-micron" RDX was also investigated.

CONCLUSION

It is concluded that the Microscopic Method given in this report is suitable for the determination of particle (crystal) size distribution of "2-micron" RDX.

It is further concluded that the Andreasen Pipette Method for particle size distribution is not suitable for the particle (crystal) size distribution of "2-micron" RDX since the dispersed particles consist of glomerates of crystals and not individual crystals.

RECOMMENDATIONS

It is recommended that the Microscopic Method be used for particle (crystal) size distribution of "2-micron" RDX and other material having this particle size distribution. It is further recommended that consideration be given to the inclusion of the method in the applicable specifications whenever such a method is needed.

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INTRODUCTION:

1. In connection with the preparation of "2-micron" RDX to be used in high velocity, low flame temperature propellants it became necessary to develop a method for the determination of the crystal size distribution of "2-micron" RDX. In this report, the expressions "particle (crystal) size distribution" and "crystal size distribution" are used interchangeably to refer to the particle size distribution of discrete crystals of RDX as distinguished from the particle size distribution of glomerates of RDX crystals. The expression "2-micron" RDX refers to a fine recrystallized grade of RDX which had been found to have an average particle diameter of 2 to 5 microns as determined by the Fisher Sub-Sieve Sizer. The sample of RDX used in this work was reported to have an average particle diameter of 4.6 microns as determined by the Fisher Sub-Sieve Sizer.

2. In conjunction with the Microscopic Method developed, attempts were made to determine the crystal size distribution of the RDX by the Andreasen Pipette Method with the aid of such dispersing agents as Marasperse CB, Marasperse N, and Marasperse C[a] since it was considered probable that the Andreasen Pipette Method would prove satisfactory if a suitable dispersing agent could be found. Preliminary work indicated that Marasperse CB was apparently the most effective of the three dispersing agents. This dispersing agent was, therefore, used in the investigation of the method.

Historical Background:

3. A thorough survey of methods of determining particle size distribution has been prepared by B. T. Federoff et al (Reference F). Since this work will shortly be published it is not considered desirable to duplicate that material in this report. It should be noted, however, that the Andreasen Pipette Method was first described in 1929 (Reference G). Microscopic Methods for particle size distribution have previously been described in Reference A and H.

Theoretical Background:

4. In a spherical particle, every diameter of the particle is always equal, regardless of the point of origin of the diameter. However, since most particles encountered in particle size measurements are irregular in shape, the question of what parameters should be established to define the size of particles arises. G. Allen Cave et al (Reference C) measure the longest, second longest and shortest dimension of each particle from which they calculate a mean volume, a mean particle diameter, a mean area, a ratio of longest to shortest dimension and distribution of ratios of longest to shortest dimension. These criteria are of value where the shape distribution are required. However, for statistical purposes when only the particle size distribution is desired, we may substitute for the irregular particle

a - Marasperse Dispersing Agents are products of Marathon Corporation, Chemical Division, Rothschild, Wisconsin

an equivalent sphere, the size of which may be defined by a single parameter, i.e., the distance between the extreme limits of a particle along a fixed direction (Reference D). Martin, et al (References A and E) explains this type of statistical measurements in the following manner. If 1000 irregular particles of precisely the same apparent diameter are selected at random and placed in a container, then these particles would form a grade whose statistical diameter is called "d". As the 1000 particles lie in all sorts of haphazard directions, their diameters are measured in the long run with equal frequency in every direction and therefore may be considered as replaceable by 1000 sperical particles of the same diameter "d". (Statistically, it may be established that for the particle size distribution encountered in "2-micron" RDX only 200 measurements of "d" are required to obtain a statistical distribution (See Discussion)).

5. The microscopic measurement of a particle size distribution is a direct method as distinguished from an indirect method such as the sedimentation methods which depend upon Stokes' law for a falling body. In the sedimentation methods, of which the Andreasen Pipette Method is an example, the time required for the particle to pass two fixed points in a suitable liquid medium is observed. Since Stokes' law relates the diameter of the falling particle to its velocity in any given medium, an "effective" diameter may be calculated for the particle. This "effective" diameter is the diameter of a sphere which takes the same time to fall a given distance as does the irregular particle. The "effective" diameter has found widespread usage in sedimentation studies. However, in addition to difficulties in its determination, such as the problem of an adequate dispersion, it should be noted that the "effective" diameter is influenced by the configuration and condition of the particle surfaces. It is, therefore, possible for two particles having the same nominal diameter and density to have different effective diameters (Reference D).

RESULTS:

6. A summary of the results obtained by the Microscopic Method is as follows (see Table I through VI):

Number of Measurements	Average Crystal Size ($\bar{x}$), microns	Standard Deviation, microns
100	3.06	1.46
200	2.83	1.30
300	2.73	1.32
400	2.64	1.30
500	2.52	1.25

7. A tabulation of the results obtained by the Andreasen Pipette Method is given in Table VIII.

8. In connection with the Andreasen Pipette Method photomicrographs, M-41054/1, M-41054/2, M-41054/3, M-41054/4, M-41054/5, M-41054/6, M-41054/7, M-41054/8, and M-41054, illustrating the appearance of the RDX remaining in suspension after the calculated time interval for the settling of each size fraction of RDX have been included in this report.

DISCUSSION OF RESULTS:

9. Since the particle size distribution of "2-micron" RDX obtained by the Andreasen Pipette Method is of a considerably greater magnitude than the particle (crystal) size distribution obtained by the Microscopic Method (compare Table I through VI with Table VIII) it is inferred that complete dispersion of the RDX crystals are not attained in the Andreasen Pipette Method. This lack of complete dispersion is confirmed by the attached photomicrographs. The photos also show that the larger size glomerates settle during the initial time intervals resulting in a false crystal size distribution.

10. Although 5 fields of 100 crystals each were measured by the microscopic method in order to evaluate the method, the standard deviation obtained for the measurements of 2 fields of 100 crystals each indicate that 200 measurements are sufficient to give a representative sample of the population. This may be concluded from the fact that the standard deviation becomes practically constant after 200 measurements of crystal size and is confirmed by Reference A. For this reason the "Microscopic Method" given under "Experimental Procedure" requires that only 200 measurements be made.

11. From the information gained in the development of the "Microscopic Method" it is indicated that the method requires approximately one hour of working time and one (1) hour of elapsed time for one (1) determination.

12. The statistical treatment of data practiced in this report is in accordance with the method described in Reference B.

EXPERIMENTAL PROCEDURE:

Microscopic Method:

13. Scope - The Microscopic Method for the particle (crystal) size distribution of "2-micron" RDX has been developed for RDX having an average crystal diameter of approximately 2.8 microns and standard deviation of 1.3 microns. The method is considered applicable to other materials having approximately this distribution but should not be used for materials in which the particle size distribution is more widely dispersed unless a much larger number of measurements is made.

14. Apparatus - Use a microscope equipped with a Filar type micrometer eye piece and such an objective (approximately 43X) that the total magnification of the optical system is approximately 550X. The filar type micrometer

eyepiece (see Note 1) consists of a micrometer screw which acts on a slide carrying a movable wire which moves in a verticle plane across the field under observation. The scale on the handle of the micrometer screw must be accurately divided into 100 equal graduations. The micrometer eyepiece is equipped with a fine fixed line running through the center of the field, parallel to the axis of the screw, to serve as a guide in orienting the object with reference to the direction of motion of the movable wire. A scale placed in the field and ruled in intervals of 0.5 mm serves for counting the revolutions of the screw. Every second interval of the scale is numbered.

The eyepiece must have a magnification of approximately 12.5X and must fit securely into the tube of the microscope.

15. Preparation of Specimen Slides - Transfer approximately 1 gram of sample to a porcelain filtering crucible of very fine porosity (Selas No. 3001 or equivalent). Wash the sample with three 20 ml portions of absolute alcohol and aspirate the sample until the odor of alcohol can no longer be detected. Transfer a small portion of the dry sample about the size of a pinhead to a glass slide. Disperse the sample on the slide in one drop of paraffin oil with the aid of a rubber policeman and cover the sample with a glass cover slip.

16. Determination of Particle (Crystal) Size Distribution - Measure the distance between opposite sides of each of 100 crystals to the nearest 0.1 unit in a field expressed in terms of divisions on the Filar micrometer scale, taking crystals near the center of the field. When measuring crystals in an aggregate or flocculate, measure every crystal in the group; in going from one group of crystals to another, measure the individual crystals between the two groups. Do not attempt to select crystals for measurements on a basis of personal judgement of the apparent distribution of the field. Repeat the above procedure for 100 crystals in another field of the slide. Calculate the size of the individual crystals from the following formula:

$$\text{Size of crystal in microns} = \frac{A}{B}$$

where:

A = distance between opposite sides of the crystal expressed in terms of divisions on the filar micrometer scale

B = Number of divisions on the filar micrometer scale equivalent to one micron

Prepare a table similar to Table 5. In the column labeled cell boundaries, insert the cell boundaries shown in Table 5.

Note 1: A Filar micrometer eyepiece, Bausch and Lomb, Cat No. 6867, Arthur H Thomas Co, Phila, Pa. has been found satisfactory for this purpose; however, it must be accurately calibrated against a stage micrometer graduated in 0.01 mm divisions. A stage micrometer, Bausch and Lomb, Cat No. 6850-A, Arthur H. Thomas Co, Phila, Pa. has been found suitable for this purpose.

The cell boundaries represent the upper and lower limits of each size group in ascending order. If crystals are found which are larger than 9.95 microns continue the listing of cell boundaries to include the larger crystals. In the column labeled cell midpoint record the midpoint of each cell. In the column labeled frequency, record the number of crystals found in each cell or size group. Estimate the cell in which the particle of average size would fall and in the column labeled "Deviation in cells from Assumed Origin", insert a zero for that cell. Consider the cell marked zero to be the origin of an arithmetical series in which the deviation of each cell is represented by the series N_s..... -3, -2, -1, 0, +1, +2, +3, +4 ..... N_l where N_s represents the deviation from the assumed origin of the cell representing the smallest particles and N_l represents the deviation from the assumed origin of the cells representing the largest particles. In the column labeled fd, record the product of the frequency and deviation from the assumed origin for each cell indicating whether the product is positive or negative. In the column labeled fd^2, record the product of the frequency and the square of the deviation from the assumed origin for each cell. On the line labeled summation, record the total number of observations in the column labeled "frequency", the algebraic sum of the values in the column fd and the sum of the values in the column fd^2. Calculate (1) the average crystal size in microns according to formula 1 and (2) the standard deviation in microns according to formula 2.

Calculations:

Formula 1 - Average particle (crystal) size ($\bar{X}$) (microns) = $A \pm \frac{0.5 \Sigma fd}{N}$

where:

A = midpoint of assumed origin

Σfd = algebraic sum of the values in the column fd

N = number of measurements

Formula 2 - Standard Deviation = $0.5 \sqrt{\frac{\Sigma fd^2}{N} - \left(\frac{\Sigma fd}{N}\right)^2}$

where:

Σfd^2 = sum of the values in the column fd^2

Σfd = sum of the values in the column fd

N = number of measurements

Andreasen Pipette Method:

17. Apparatus:

Assembly - The Andreasen Pipette Assembly consists of a sedimentation cylinder into which a pipette with a ground fitting is inserted. The lower end of the pipette has an internal diameter of 1.1 ± 0.1 mm and extends to within 3.8 ± 0.1 cm of the bottom of the sedimentation cylinder. The pipette is equipped with a two way stopcock in order that liquid can be drawn up through the lower end of the pipette and stopcock into a bulb, having a nominal capacity of 10 ml, and can be discharged from the bulb through the hollow center of the barrel of the stopcock. The upper portion of the pipette extends approximately 10 cm above the bulb and is connected by means of a glass "Y" tube and rubber fitting to a small reservoir which contains liquid for rinsing the bulb. A rubber tube is connected to the third arm of the "Y" tube for the application of suction when the bulb is being filled with liquid from the sedimentation cylinder. The flow of liquid for rinsing from the reservoir is controlled by a pinch-clamp attached to the rubber tube. The pipette is accurately calibrated in order that the amount of liquid delivered by the pipette when it is drained will be 10 ml. The sedimentation cylinder of the Andreasen Pipette Assembly has an internal diameter of 5.5 ± 0.5 cm and is 29.0 ± 1.0 cm high. It is accurately graduated in cms to a height of 20 cm from the lower end of the pipette. To draw liquid up from the sedimentation cylinder into the bulb of the pipette, adjust the stopcock, apply suction via the rubber tube and suck the liquid up into the bulb to the 10 ml mark. Discontinue the suction and close the stopcock.

Crucibles - Use porcelain, sintered bottom filtering crucible of very fine porosity (Selas No. 3001 or equivalent).

Filter Flasks - Use vacuum type filtering flasks of 100 ml capacity.

Dispersion Tube - The dispersion tube is 5 inches long, has an internal diameter of 5/8 inch, and is equipped with a ground glass stopcock at the bottom.

Dispersion Brush - The dispersion brush is approximately 2 inches long, with a tufted tip and fits snugly into the dispersion tube.

Motor - Use a heavy-duty motor equipped with a suitable chuck to grip the wire end of the brush. The Motor must have a minimum rating of 900 RPM.

18. Procedure:- Add approximately 7 ml of the dispersion solution consisting of approximately 0.5 gram of Marasperse, CB per 100 ml water. Transfer an accurately weighed 10 gm portion of sample to the dispersion

tube. Insert the brush in the dispersion tube and work the brush up and down to disperse the sample throughout the liquid. Push the brush down into the tube until the tuft of the brush is in contact with the base of the tube. Attach the wire handle of the brush to the chuck of the motor. Fasten the dispersion tube to the stand with the aid of a clamp. The lower half of the tube is immersed in a beaker of cold water to prevent over heating as the suspension is stirred by the brush. Turn the motor on and stir the suspension for 15 minutes at a rate of 900 RPM. Turn the motor off and quantitatively transfer the suspension to the sedimentation cylinder of the Andreasen Pipette. Add sufficient dispersing solution to the cylinder to bring the liquid to the mark when the pipette is in position. (Adjust the temperature of the suspension to 25.0°C.) Shake the Andreasen Pipette Assembly for approximately 2 minutes and place the assembly in a constant temperature bath maintained at 25.0°C. At the end of each time interval calculated for the desired particle size range, described under calculations, withdraw a 10 ml aliquot of the suspension of RDX from the sedimentation cylinder by sucking it up into the bulb of the pipette to the mark. Transfer the contents of the bulb to a tared filtering crucible attached to a 100 ml filtering flask and aspirate. Rinse the bulb of the Andreasen pipette with 3, three ml portions of dispersing solution from the reservoir, quantitatively transfer each portion of solution to the filtering crucible and aspirate. Wash the contents of the crucible with three, five ml portions of distilled water and two, five ml portions of ethyl alcohol. Dry the crucible in an oven at 105° ± 5°C for 10 minutes, cool in a desiccator and weigh.

19. Calculation for Andreasen Pipette Method:

Calculation of time required for specified fraction -

$$t = \frac{Kn}{d^2}$$

where:

$$K = \frac{3.061 \times 10^4}{D_1 - D_2} \cdot V = 382.6$$

t = Time from start of test in minutes

h = Distance in centimeters from the surface of the suspending medium to the tip of the Andreasen pipette when the aliquot is withdrawn

d = Diameter, in microns of a sphere falling at the same rate as the particle

D_1 = 1.816 (density of particle, in grams per cc)

D_2 = 1.000 (density of suspending medium, in grams per cc

V = 0.0102 (viscosity of suspending medium in poises)

Calculation of Percentage of Sample Finer than Specified Diameter -

$$P = \frac{100\ C}{W}$$

where:

P = percent of sample finer than specified diameter
C = gain in weight of crucible after filtration
W = calculated weight of suspended particles in aliquot before sedimentation occurs

REFERENCES:

A - Micromeritics by J. M. Dallavalle, p 60, (1943) Pitman Publishing Company, New York

B - Statistical Quality Control by E. L. Grant, p 66, 1st Ed, (1946) McGraw-Hill Book Company, Inc, New York

C - Cave, G. Allen, Krotinger, Nathan J. and McCalet, John D., Industrial and Engineering Chemistry, Volume 41, pp 1286-90, (1949)

D - Micromeritics by J. M. Dallavalle, pp 31-32, (1943) Pitman Publishing Company, New York

E - Martin, Geoffrey, et al, Trans Brit, Ceram Soc, Volume 23, pp 61-109 (1923)

F - Dr. B. T. Federoff et al, unpublished work

G - Andreasen, A.H.M., Kolloid Xtg, Volume 49, No. 48, p 252 (1929) and Zement, Volume 19, p 698 (1930)

H - "Photomicrography in Theory and Practice" by C. P. Shillaber, pp 300 and 725, J. Wiley and Sons, New York

INCLOSURES:

Tables I through VIII
9 Photomicrographs

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TABLE I

Crystal Size Distribution by Microsconic Measurement

Cell Boundaries Microns [i]	Cell Midpoint Microns [a]	Field 1 Frequency [b]	Field 2 Frequency [b]	Field 3 Frequency [b]	Field 4 Frequency [b]	Field 5 Frequency [b]	Total Frequency [b]	Average Frequency Percent
0.45 to 0.95	0.7	0	1	2	0	4	7	1.4
0.95 to 1.45	1.2	1	10	15	18	20	64	12.8
1.45 to 1.95	1.7	26	22	29	28	35	139	27.8
1.95 to 2.45	2.2	20	21	17	23	18	99	19.8
2.45 to 2.95	2.7	8	12	6	9	10	45	9.0
2.95 to 3.45	3.2	14	12	11	7	4	48	9.6
3.45 to 3.95	3.7	10	13	6	2	4	35	7.0
3.95 to 4.45	4.2	4	2	6	4	3	19	3.8
4.45 to 4.95	4.7	4	4	2	4	0	14	2.8
4.95 to 5.45	5.2	5	1	3	3	2	14	2.8
5.45 to 5.95	5.7	4	1	2	1	0	8	1.6
5.95 to 6.45	6.2	3	1	1	0	0	5	1.0
6.45 to 6.95	6.7	0	0	0	1	0	1	0.2
6.95 to 7.45	7.2	0	0	0	0	0	0	0.0
7.45 to 7.95	7.7	0	0	0	0	0	0	0.0
7.95 to 8.45	8.2	0	0	1	0	0	1	0.2
8.45 to 8.95	8.7	0	0	0	0	0	0	0.0
8.95 to 9.45	9.2	0	0	0	0	0	0	0.0
9.45 to 9.95	9.7	1	0	0	0	0	1	0.2
								100.0
Number of Observations		100	100	100	100	100	500	
Average crystal size, microns		3.1	2.6	2.4	2.4	2.0		

Notes: See notes a, b, and i in Table VII.

TABLE II

Crystal Size Distribution by Microscopic Measurement

Computation of Average crystal size and Standard Deviation Frequency Distribution for 100 Measurements

Cell Boundaries Microns [i]	Cell Midpoint Microns [a]	f Frequency [b]	d Deviation in cells from assumed origin [c]	fd [d]	fd^2 [e]
0.45 to 0.95	0.7	0	-4	0	0
0.95 to 1.45	1.2	1	-3	-3	9
1.45 to 1.95	1.7	26	-2	-52	104
1.95 to 2.45	2.2	20	-1	-20	20
2.45 to 2.95	2.7	8	0	0	0
2.95 to 3.45	3.2	14	+1	+14	14
3.45 to 3.95	3.7	10	+2	+20	40
3.95 to 4.45	4.2	4	+3	+12	36
4.45 to 4.95	4.7	4	+4	+16	64
4.95 to 5.45	5.2	5	+5	+25	125
5.45 to 5.95	5.7	4	+6	+24	144
5.95 to 6.45	6.2	3	+7	+21	147
6.45 to 6.95	6.7	0	+8	0	0
6.95 to 7.45	7.2	0	+9	0	0
7.45 to 7.95	7.7	0	+10	0	0
7.95 to 8.45	8.2	0	+11	0	0
8.45 to 8.95	8.7	0	+12	0	0
8.95 to 9.45	9.2	0	+13	0	0
9.45 to 9.95	9.7	1	+14	+14	196
Summation (Σ, f		100		+91	899

Average crystal size ($\bar{X}$) in microns [g] = 3.06

Standard deviation in microns [h] = 1.46

See notes [a] through [i] in Table VII.

TABLE III

Crystal Size Distribution by Microscopic Measurement

Computation of Average Crystal Size and Standard Deviation of Frequency Distribution for 200 Measurements

Cell Boundaries Microns [i]	Cell Midpoint Microns [a]	f Frequency [b]	d Deviation in Cells from assumed origin [c]	fd [d]	fd^2 [e]
0.45 to 0.95	0.7	1	-4	-4	16
0.95 to 1.45	1.2	11	-3	-33	99
1.45 to 1.95	1.7	48	-2	-96	192
1.95 to 2.45	2.2	41	-1	-41	41
2.45 to 2.95	2.7	20	0	0	0
2.95 to 3.45	3.2	26	+1	+26	26
3.45 to 3.95	3.7	23	+2	+46	92
3.95 to 4.45	4.2	6	+3	+18	54
4.45 to 4.95	4.7	8	+4	+32	128
4.95 to 5.45	5.2	6	+5	+30	150
5.45 to 5.95	5.7	5	+6	+30	180
5.95 to 6.45	6.2	4	+7	+28	196
6.45 to 6.95	6.7	0	+8	0	0
6.95 to 7.45	7.2	0	+9	0	0
7.45 to 7.95	7.7	0	+10	0	0
7.95 to 8.45	8.2	0	+11	0	0
8.45 to 8.95	8.7	0	+12	0	0
8.95 to 9.45	9.2	0	+13	0	0
9.45 to 9.95	9.7	1	+14	14	196
Summation (Σ) [f]		200		+50	1370

Average crystal size ($\overline{X}$) in microns [g] = 2.83
Standard deviation (Sigma) in microns [h] = 1.30

TABLE IV

Crystal Size Distribution by Microscopic Measurement

Computation of Average Crystal Size and Standard Deviation of Frequency Distribution for 300 Measurements

Cell Boundaries Microns [i]	Cell Midpoint Microns [a]	f Frequency [b]	d Deviation in Cells From Assumed Origin [c]	fd [d]	fd^2 [e]
0.45 to 0.95	0.7	3	-4	-12	48
0.95 to 1.45	1.2	26	-3	-78	234
1.45 to 1.95	1.7	76	-2	-152	304
1.95 to 2.45	2.2	58	-1	-58	58
2.45 to 2.95	2.7	26	0	0	0
2.95 to 3.45	3.2	37	+1	+37	37
3.45 to 3.95	3.7	29	+2	+58	116
3.95 to 4.45	4.2	12	+3	+36	108
4.45 to 4.95	4.7	10	+4	+40	160
4.95 to 5.45	5.2	9	+5	+45	225
5.45 to 5.95	5.7	7	+6	+42	252
5.95 to 6.45	6.2	5	+7	+35	245
6.45 to 6.95	6.7	0	+8	0	0
6.95 to 7.45	7.2	0	+9	0	0
7.45 to 7.95	7.7	0	+10	0	0
7.95 to 8.45	8.2	1	+11	+11	121
8.45 to 8.95	8.7	0	+12	0	0
8.95 to 9.45	9.2	0	+13	0	0
9.45 to 9.95	9.7	1	+14	+14	196
Summation (Σ) [f]		300		+18	2104

Average crystal size ($\bar{X}$) in microns [g] = 2.73

Standard deviation (Sigma) in microns [h] = 1.32

TABLE V

Crystal Size Distribution by Microscopic Measurement

Computation of Average Crystal Size and Standard Deviation of Frequency Distribution for 400 Measurements

Cell Boundaries, Microns i	Cell Midpoint, microns a	f Frequency b	d Deviation in Cells from Assumed Origin c	fd d	fd^2 e
0.45 to 0.95	0.7	3	-4	- 12	48
0.95 to 1.45	1.2	44	-3	-132	396
1.45 to 1.95	1.7	104	-2	-208	416
1.95 to 2.45	2.2	81	-1	- 81	81
2.45 to 2.95	2.7	35	0	0	0
2.95 to 3.45	3.2	44	∓1	∓ 44	44
3.45 to 3.95	3.7	31	∓2	∓ 62	124
3.95 to 4.45	4.2	16	∓3	∓ 48	144
4.45 to 4.95	4.7	14	∓4	∓ 56	224
4.95 to 5.45	5.2	12	∓5	∓ 60	300
5.45 to 5.95	5.7	8	∓6	∓ 48	288
5.95 to 6.45	6.2	5	∓7	∓ 35	245
6.45 to 6.95	6.7	1	∓8	∓ 8	64
6.95 to 7.45	7.2	0	∓9	0	0
7.45 to 7.95	7.7	0	∓10	0	0
7.95 to 8.45	8.2	1	∓11	∓ 11	121
8.45 to 8.95	8.7	0	∓12	0	0
8.95 to 9.45	9.2	0	∓13	0	0
9.45 to 9.95	9.7	1	∓14	∓ 14	196
Summation $(\Sigma)^f$		400		- 47	2691

Average crystal size $(\bar{X})$ in microns g = 2.64

Standard deviation (Sigma) in microns h = 1.30

TABLE VI

Crystal Size Distribution by Microscopic Measurement

Computation of Average Crystal Size and Standard Deviation of Frequency Distribution of 500 Measurements

Cell Boundaries, microns i	Cell Midpoint micron a	f Frequency b	Deviation in Cells From Assumed Origin c	fd a	fd^2 e
0.45 to 0.95	0.7	7	- 4	- 28	112
0.95 to 1.45	1.2	64	- 3	-192	576
1.45 to 1.95	1.7	139	- 2	-278	556
1.95 to 2.45	2.2	99	- 1	- 99	99
2.45 to 2.95	2.7	45	0	0	0
2.95 to 3.45	3.2	48	∕ 1	∕ 48	48
3.45 to 3.95	3.7	35	∕ 2	∕ 70	140
3.95 to 4.45	4.2	19	∕ 3	∕ 57	171
4.45 to 4.95	4.7	14	∕ 4	∕ 56	224
4.95 to 5.45	5.2	14	∕ 5	∕ 70	350
5.45 to 5.95	5.7	8	∕ 6	∕ 48	288
5.95 to 6.45	6.2	5	∕ 7	∕ 35	245
6.45 to 6.95	6.7	1	∕ 8	∕ 8	64
6.95 to 7.45	7.2	0	∕ 9	0	0
7.45 to 7.95	7.7	0	∕10	0	0
7.95 to 8.45	8.2	1	∕11	∕ 11	121
8.45 to 8.95	8.7	0	∕12	0	0
8.95 to 9.45	9.2	0	∕13	0	0
9.45 to 9.95	9.7	1	∕14	∕ 14	196
Summation (Σ) f		500		-180	3190

Average crystal size ($\bar{X}$) in microns f = 2.52

Standard Deviation (Sigma) in microns h = 1.25

TABLE VII

Key to Tables I-VI

a The cell midpoint is obtained by taking the sum of the upper and lower cell boundaries and dividing by 2.

b The frequency is the number of crystals found in each statistical cell.

c The deviation in cells from the assumed origin is obtained by estimating the cell in which the crystal of average size would fall and assigning a value of zero to that cell, thereby assuming that cell to be the origin. The deviation of the adjacent cell representing the next smaller group of crystals is assigned a deviation value of -1 and the next cell representing smaller crystals is assigned a deviation value of -2, etc. In like manner the cell adjacent to the assumed origin and representing the next larger size-group of crystals is assigned a deviation value of +1 and the next cell representing the next larger size-group of crystals is assigned a deviation value of +2, etc. The deviation in cells from the assumed origin is therefore represented by the arithmetical series $-N_s \ldots\ldots -3, -2, -1, 0, +1, +2, +3 \ldots\ldots N_L$ where N_s is the deviation from the assumed origin of the cell representing the smallest cyrstals and N_L is the deviation from the assumed origin of the cell representing the largest crystals.

d Obtained by multiplying the frequency (f) by the deviation (d) in cells from the assumed origin.

e Obtained by multiplying the frequency (f) by the square of the deviation (d).

f Summation is represented by the symbol Σ and is the algebraic sum of a group of values.

g Represents the average particle size (designated by $\bar{X}$) and is obtained by algebraically adding the midpoint of the assumed origin to the product of 0.5 and the average deviation in cells from the assumed origin. Thus average crystal size - $A + 0.5 \frac{(\Sigma fd)}{N}$, where A = midpoint of assumed origin, Σ fd = summation of fd, and N = the number of measurements.

h The standard deviation is calculated from the formula:

$$\text{Standard Deviation} = 0.5\sqrt{\frac{\Sigma fd^2}{N} - \left(\frac{\Sigma fd}{N}\right)^2},$$

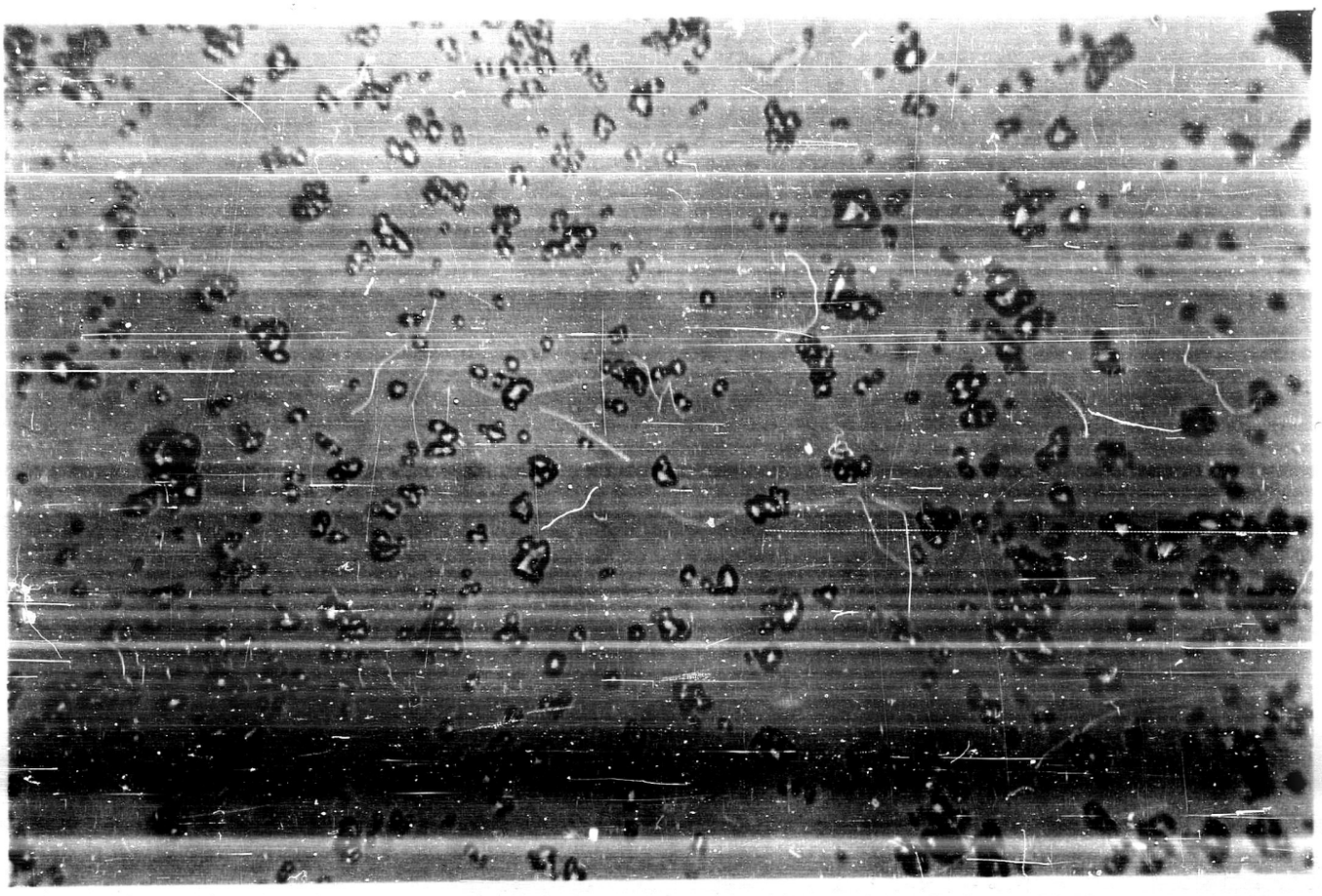
where Σfd^2 = the summation of fd^2, N = the number of measurements, and Σ fd - the summation of fd.

i The cell boundaries represent the upper and the lower limits of each size group or cell in ascending order. The cell may be defined as the interval along the scale of measurement, of each ordered class.

TABLE VIII

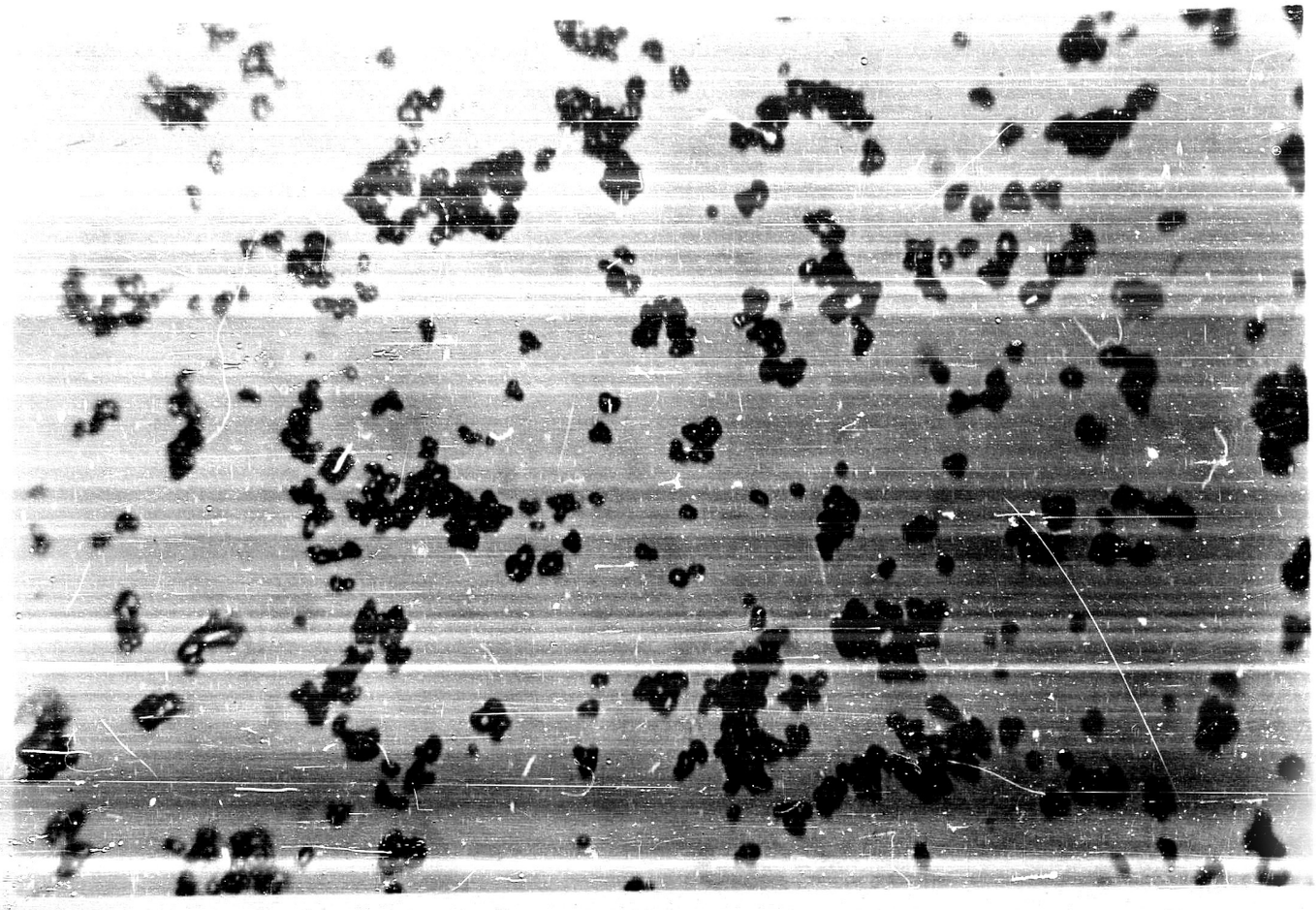
Particle Size Distribution of "2-Micron" RDX by Andreasen Pipette Method

Particle Size Range Microns	Percent RDX Found in each Range
less than 2.65	1
2.65 to 3.95	7
3.95 to 4.95	7
4.95 to 5.95	13
5.95 to 7.95	21
7.95 to 9.95	22
9.95 to 14.95	16
14.95 to 19.95	3
greater than 19.95	0



M-41054 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

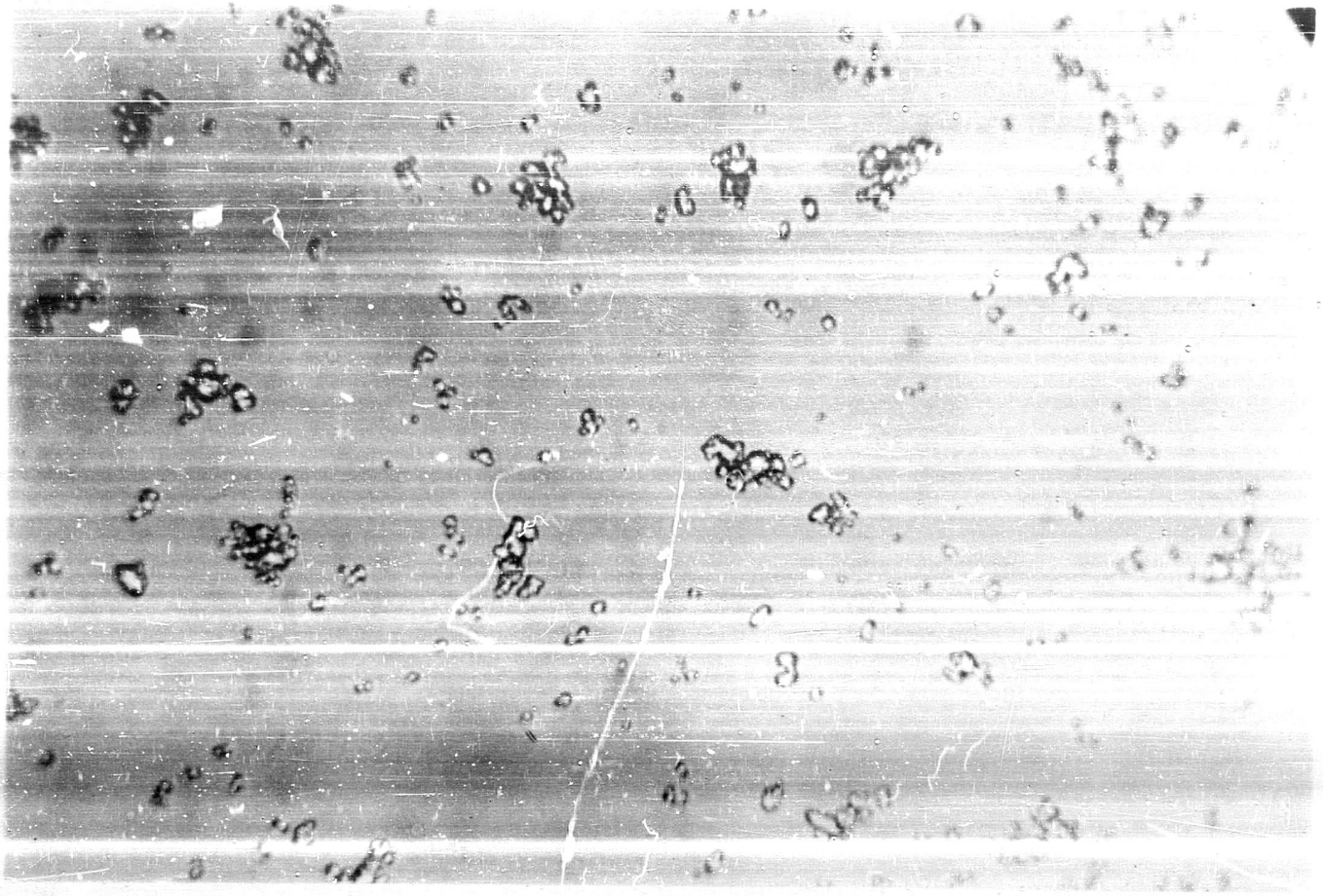
RDX (2 micron) immediately after suspension in Andreasen Pipette. Magnification 500 X



M-41054/8 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

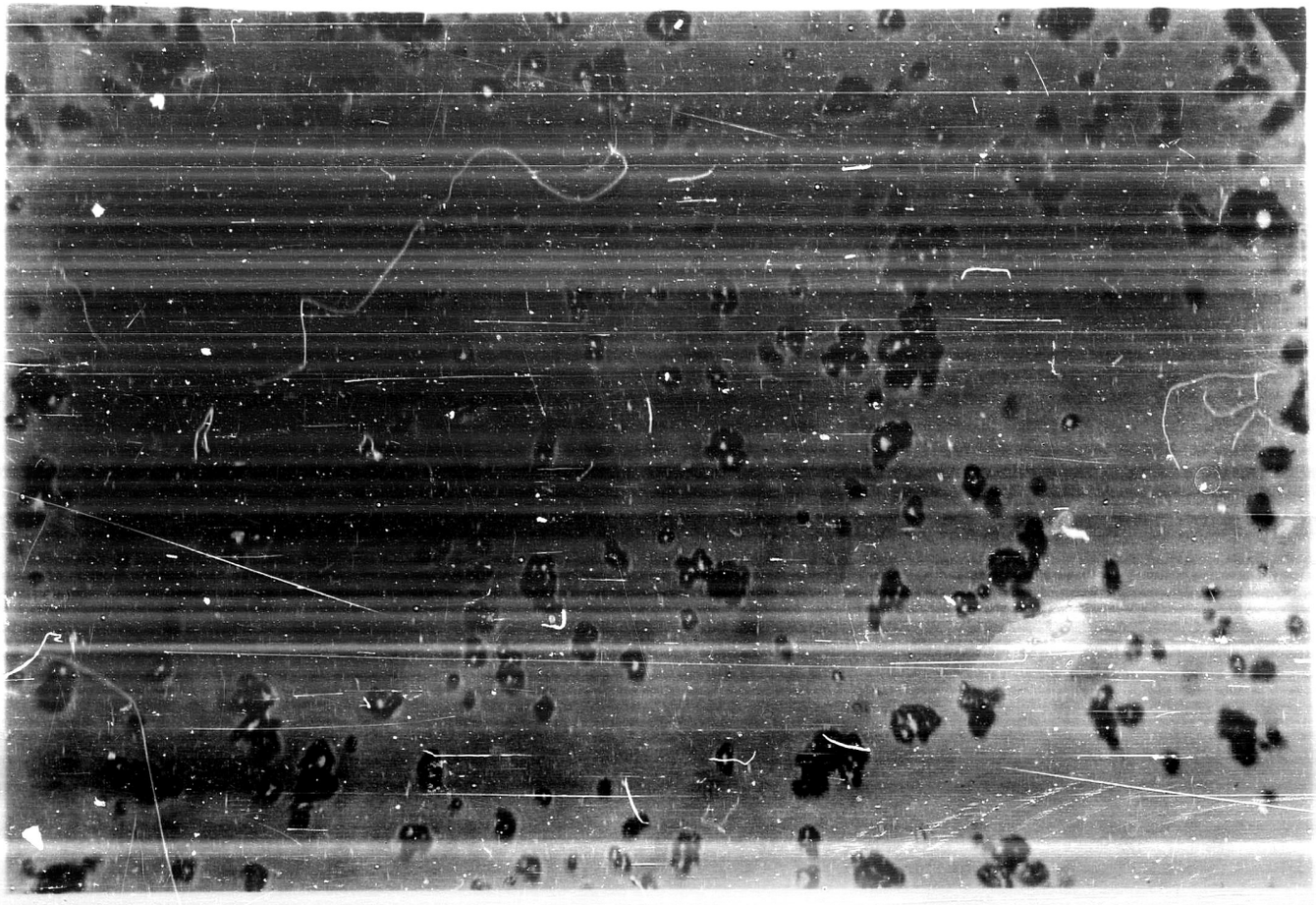
RDX (2 micron) remaining in suspension after calculated time for settling of 25 micron fraction in Andreasen Pipette.

Magnification 500 X



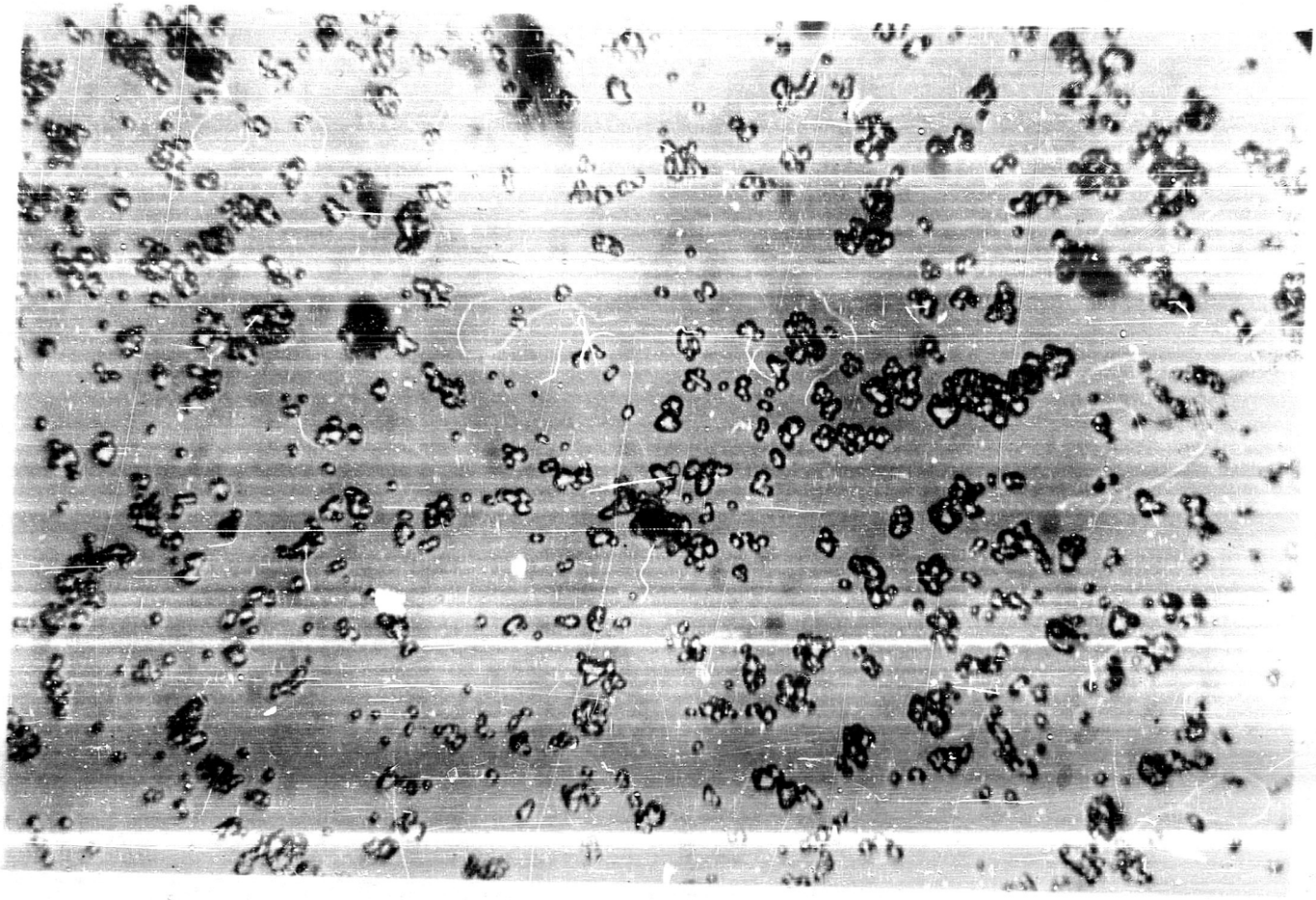
M-41054/7 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

RDX (2 micron) remaining in suspension after calculated time for settling of 20 micron fraction in Andreasen Pipette. Magnification 500 X



M-41054/6 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

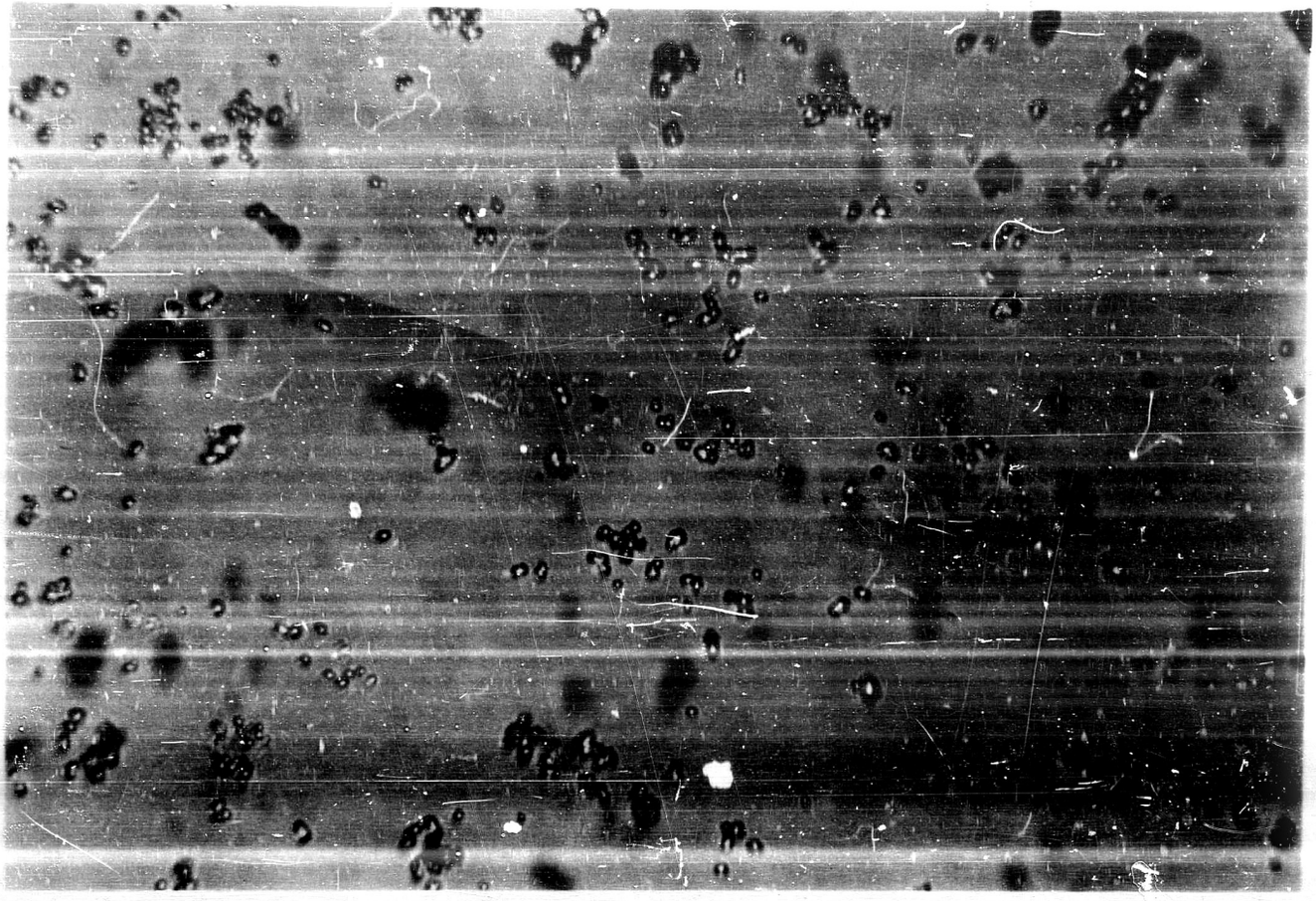
RDX (2 micron) remaining in suspension after calculated time for settling of 15 micron fraction in Andreasen Pipette. Magnification 500 X



M-41054/5 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

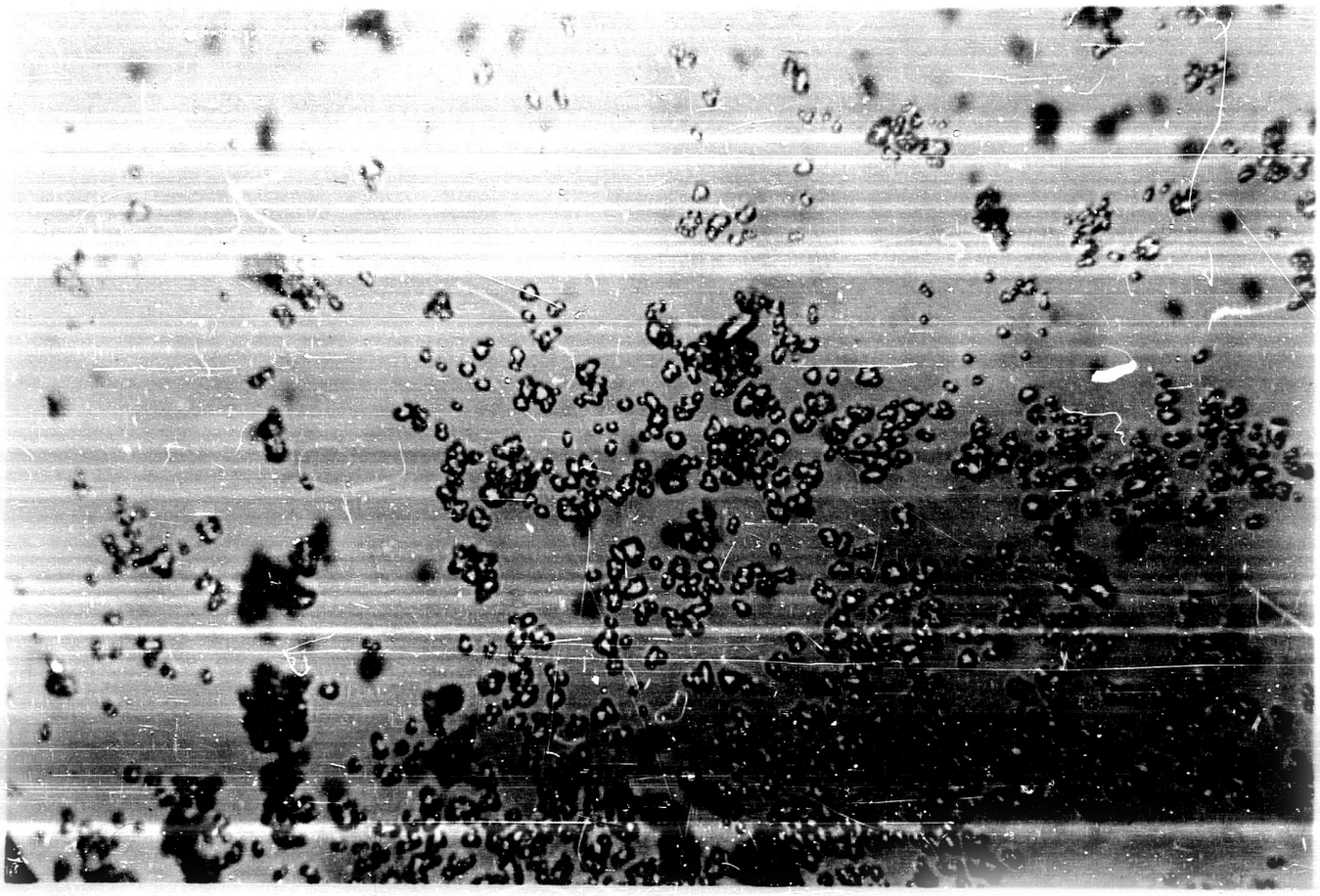
RDX (2 micron) remaining in suspension after calculated time for settling of 10 micron fraction in Andreasen Pipette.

Magnification 500 X



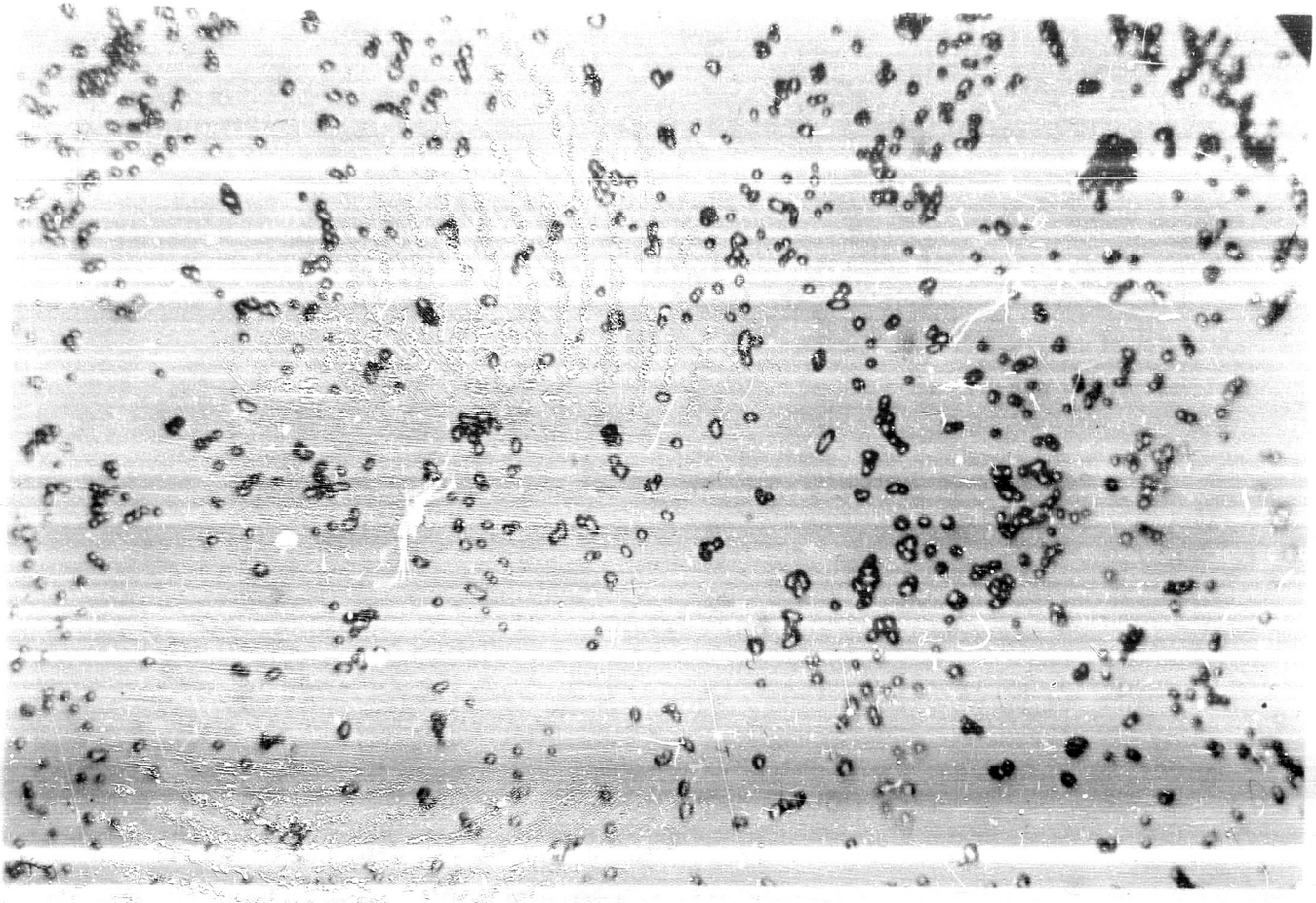
M-41054/4 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

RDX (2 micron) remaining in suspension after calculated time for settling of 8.0 micron fraction in Andreasen Pipette. Magnification 500 X



M-41054/3 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

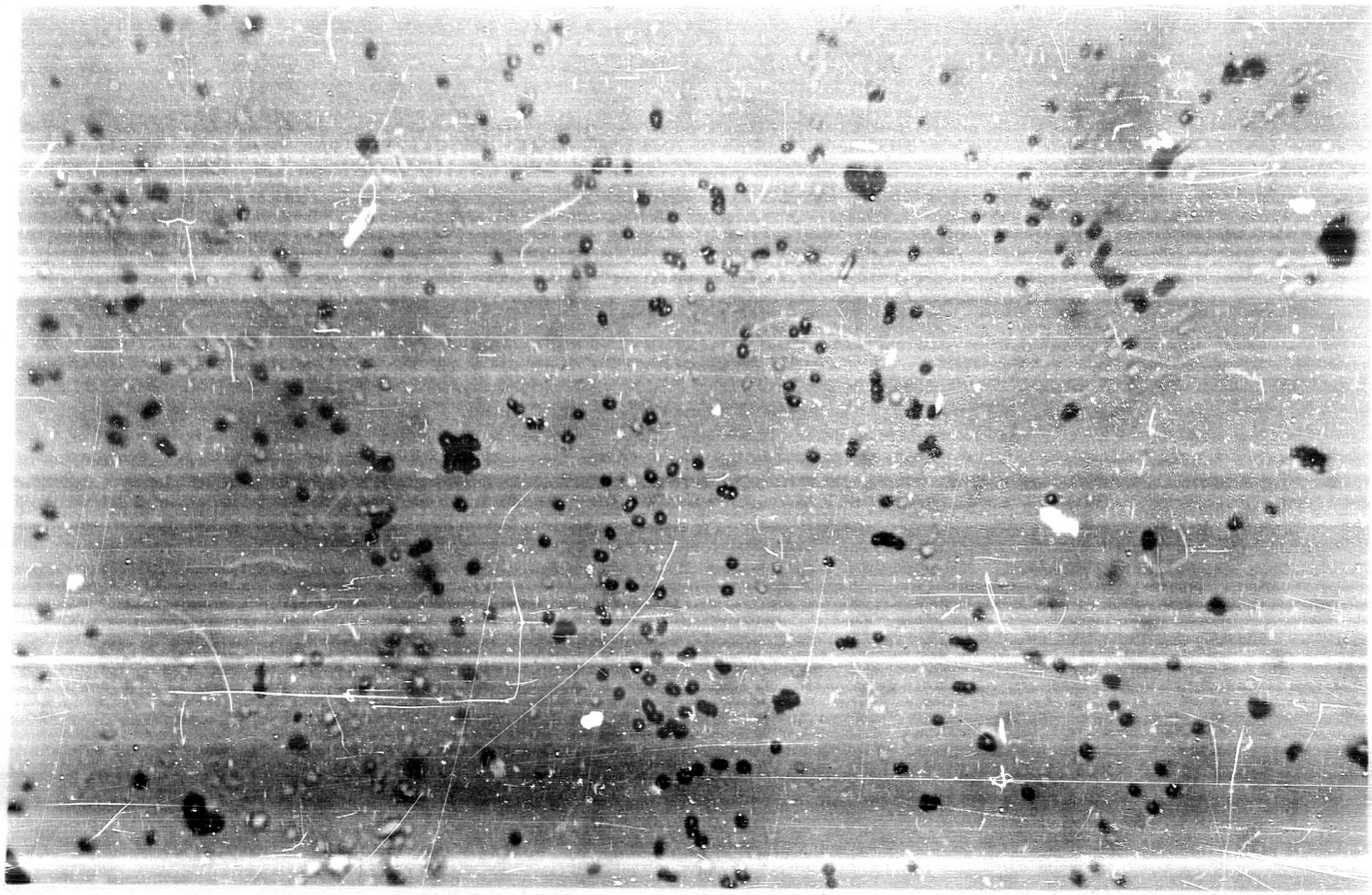
RDX (2 micron) remaining in suspension after calculated time for settling of 6.0 micron fraction in Andreasen Pipette. Magnification 500 X



M-41054/2 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

RDX (2 micron) remaining in suspension after calculated time for settling of 4.0 micron fraction in Andreasen Pipette.

Magnification 500 X



M-41054/1 April 1952 PICATINNY ARSENAL ORDNANCE CORPS

RDX (2 micron) remaining in suspension after calculated time for settling of 2.7 micron fraction in Andreasen Pipette. Magnification 500 X